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Effect of deposition parameters and the substrate condition of the crystallinity of bismuth films

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The effects of deposition rate, substrate temperature, post deposition annealing, nature of the substrate and the substrate condition on the crystallinity of vapour deposited bismuth films are studied. Films in the thickness range 50 Å, to 1000 Å deposited onto glass, mica and sodium chloride surfaces under different conditions have been studied by transmission electron microscopy and thicker films (1000 Å to 25,000 Å) on glass and mica substrates have been analysed using an X-ray diffractometer.

1. INTRODUCTION

Bismuth in the initial stages of growth exhibits two orientations with equal probabilities on sodium chloride cleavages (Komnik & Pilipenko 1971, Kosevich *et al* 1970). Suitable changes in the condensation conditions result in a predominant (001) orientation or a predominant (012) orientation (Kosevich *et al* 1970, Zavyalova & Imanov 1969, Federenko *et al* 1968). When deposited onto ((100)) cleavages of sodium chloride the orientation of bismuth films will be such that the [[100]] direction, common to all planes, will be parallel to [[100]] or [[110]] directions of the substrate surface. It has been observed by Komnik & Pilipenko (1971) that as the thickness of the deposit is increased the contributions from various orientations change. On mica, films of thickness about 300 Å show (001) orientation with addisional(011) and (012) orientations; but for thicker films (> 900 Å) the (011) orientation is absent. A change in the orientation of bismuth films deposited onto sodium chloride cleavages from (001) to (012) has been reported by Murchakova & Kozlovskii (1971) when a constant electric filed is applied parallel to the substrate surface. The results of electron microscopic and X-ray diffractometer studies on the crystallinity of vapour deposited bismuth films as a function of the deposition parameters and the substrate conditions are reported here.

2. EXPERIMENTAL

Investigations were carried out on films deposited onto substrate surfaces by evaporating pure bismuth in a residual air pressure on the order of 10^{-6} torr.

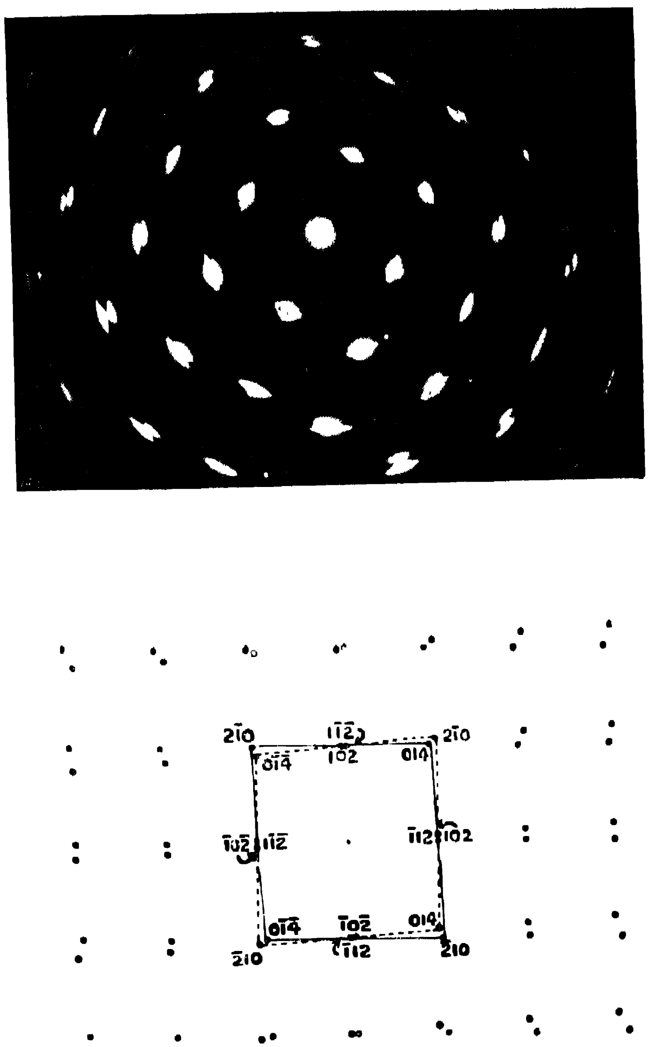
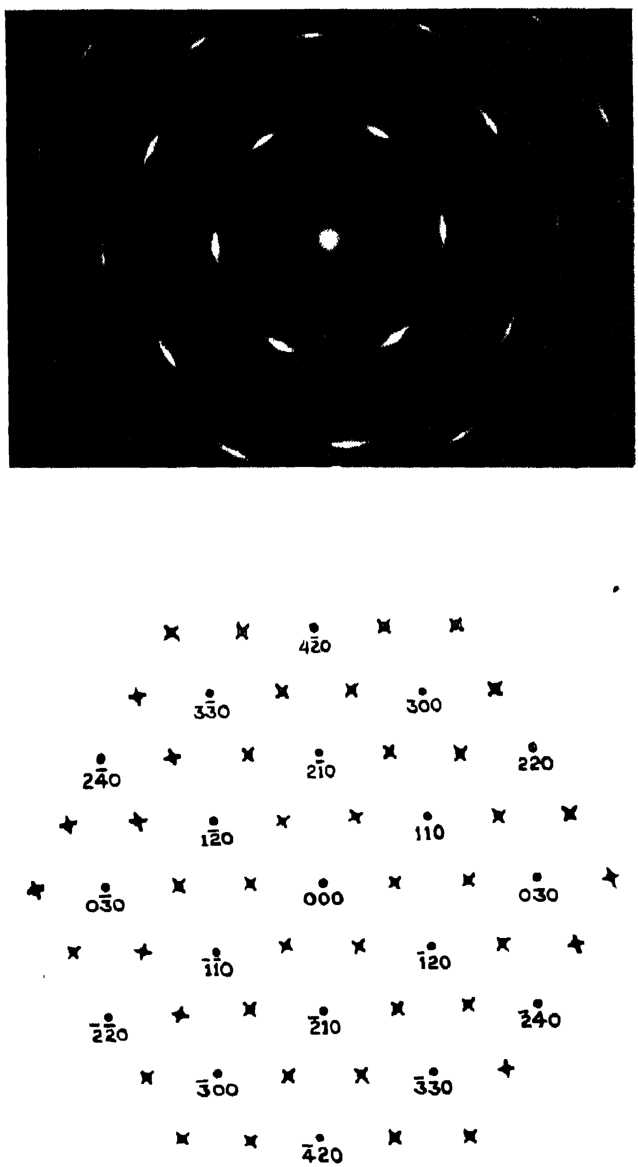
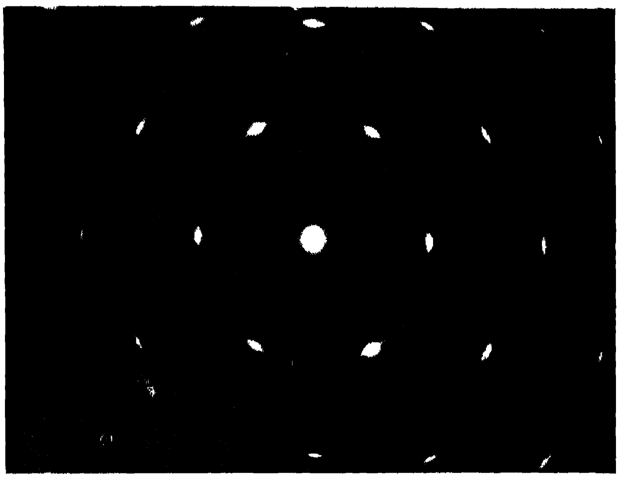


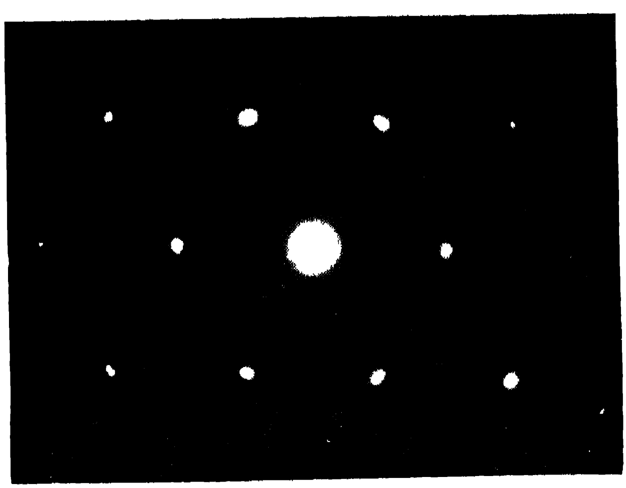
Fig. 1-4. Selected area diffraction patterns of 250 A thick bismuth films deposited onto (1) (100) NaCl at 130°C.



(2) Mica cleavage at 130°C's.



(3) Mica cleavers at 130°C and annealed at the same temperature for two hour.



(4) (111) NaCl grown on mica cleavage.

Whenever high temperature depositions were done the substrates were first heated to the required temperature in vacuum and then the films were deposited. The substrate with the film was then allowed to cool to room temperature before taking out for examination. The thicknesses of the deposits were measured interferometrically by Tolansky's method (1970). The weight thickness chart was prepared for bismuth and films of desired thickness were obtained by evaporating the required amount of the substance.

Thicker films deposited onto glass and mica surfaces were examined using an X-ray diffractometer provided with a scintillation counter detector and the diffraction patterns were recorded on an X-Y chart recorder.

3. Results

3.1. *Electron Microscopic Observations*

Bismuth films deposited onto amorphous or crystalline substrates at room temperature forms polycrystalline films It is observed that the contribution of different reflections, as revealed by the intensities of the rings, is sensitive to the deposition rate. When deposited with deposition rates in the range 5 Å sec^{-1} to 100 Å sec^{-1} the films show a mixture of (001) and (012) textures on sodium chloride substrates where as those on mica cleavages show (001) texture.

Bismith films deposited onto sodium chloride and mica cleavages at 130°C show single crystalline orientations (figures 1 and 2). The effect of deposition rate on the crystallinity of these films is considerably reduced at this deposition temperature. The films deposited at 130°C with rates varying between 5 Å sec^{-1} and 100 Å sec^{-1} were more or less identical in the sense that there was not much variation in the crystallinity of the deposits. Hence all depositions at high substrate temperatures were carried out at a rate of about 25 Å sec^{-1}. The films grown on mica show a (001) orientation whereas those on sodium chloride cleavages show (012) orientation. The diffraction patterns due to the films deposited on sodium chloride cleavages show the resultant of two azimuthal orientations perpendicular to each other as shown schematically in figure 1(b).

Post-deposition annealing of the films formed at room temperature has little effect on the crystallinity of bismuth films when the annealing temperature is below 150°C. But the films deposited at a substrate temperature 130°C when annealed at the same temperature for a couple of hours give sharp diffraction patterns (figure 3).

Substrate surfaces of sodium chloride grown in vacuum yield better single crystal films of bismuth than the air cleaved substrate surfaces. The substrate surfaces with (111) NaCl orientations were grown by depositing sodium chloride onto mica cleavages heated to 275°C as reported by Missiroli (1972). The substrates were then cooled to 130°C and bismuth was deposited onto them. The films thus grown on (111) NaCl revealed sharp diffraction patterns as the one shown in figure 4 which has been identified to be the (001) orientation.

3.2. *X-ray Analysis of Thick Bismuth Films*

Bismuth films of thicknesses 1000 Å to 25,000 Å grown on glass and mica substrates at different substrate temperatures were studied with an X-ray diffractometer. The radiation CuKα/Ni from a tube operating at 700 VA was used and the specimens were scanned in the diffraction angle 2θ range 8° to 90° at a counter speed of 1° min^{-1}.

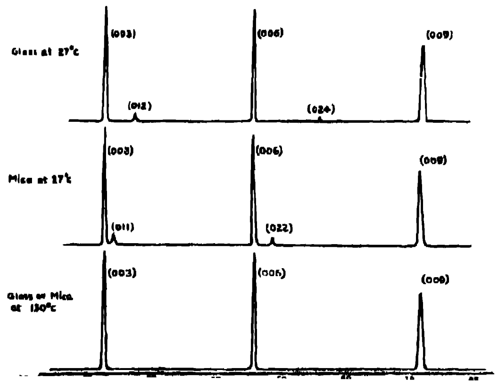


Fig. Diffractometer tracings for 1000 Å thick bismuth film on glass and mica substrat at 27°C and 130°C.

All the films deposited onto glass and mica substrates showed strong {001} texture (figure 5). The thinnest of the films (1000 A) deposited on glass at room temperature showed an additional {012} orientation and those on mica an additional {011} orientation. For all films of thickness greater than 1000 Å these orientations were very weak compared to the {001} orientation. When deposited at 130°C all the films (1000 Å to 25,000 Å thickness) on both glass surfaces and mica cleavages were purely {001} textured. The films grown on mica and glass substrates were essentially similar. in the sense that the diffraction patterns obtained were identical.

4. Conclusion

The crystallinity of bismuth films is highly influenced by the changes in the deposition parameters. But the films grown at higher substrate temperature are less affected by the variations in the deposition rate. Films grown on freshly prepared substrate surfaces show better crystallinity than those grown on substrate surfaces prepared under atmospheric conditions. Thicker films grown on amosphous glass substrates were similar in texture to those grown on isotropic mica substrates.

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REFERENCES

Burchakova V. I. & Zozlovskii M. I 1971 *Sov. Phys. Crystallogr.* **16**, 331.

Fedorenko A. I., Palatnik L. S. & Kosevich V. M. 1968 *Sov. Phys. Solid State.* **9**, 2238.

Komnik Yu F. & Pilipenko V. V 1971 *Sov. Phys. Crystallogr* **16**, 352.

Kosevich V. M., Palatnik L. S., Grigorov S. N. & Zolotnitskii Yu V. 1970 *Sov. Phys. Solid State* **11**, 2547.

Missiroli G. F. 1972 *Thin Solid Films* **12**, S35.

Tolansky S. 1970 *Multiple beam interference microscopy of metals* (Academy Press, New York) p. 95.

Zavyalova A. A. & Imanov R. M. 1969 *Sov. Phys. Crystallogr.* **14**, 305.